Journal of ASTM International October 2009. Vol. XX No. XX

Paper ID

Available online at www.astm.org

Kyle Sparks,[1] Joel M. Stoltzfus[2], Theodore A. Steinberg[3], and David Lynn[4]

Determination of Pass/Fail Criteria for Promoted Combustion Testing

ABSTRACT: Promoted ignition testing [1–3] is used to determine the relative flammability of metal rods in oxygen-enriched atmospheres. In these tests, a promoter is used to ignite each metal rod to start the sample burning. Experiments were performed to better understand the promoted ignition test by obtaining insight into the effect a burning promoter has on the preheating of a test sample. Test samples of several metallic materials were prepared and coupled to fast-responding thermocouples along their length. Various ignition promoters were used to ignite the test samples. The thermocouple measurements and test video was synchronized to determine temperature increase with respect to time and length along each test sample. A recommended length of test sample that must be consumed to be considered a flammable material was determined based on the preheated zone measured from these tests. This length was determined to be 30 mm (1.18 in.). Validation of this length and its rationale are presented.

KEYWORDS: Oxygen, ignition, temperature, burning, rods, flammability, promoted combustion, heat affected, promoter, WSTF

[1] Mechanical Engineer, NASA Test and Evaluation Contract, P.O. Box 20, Las Cruces, New Mexico, 88004.
[2] Laboratories Office, NASA Johnson Space Center White Sands Test Facility, Las Cruces, New Mexico, 88004.
[3] Senior Lecturer, School of Engineering Systems, Queensland University of Technology (QUT), Brisbane, Qld 4001, Australia.
[4] Postgraduate Student, School of Engineering Systems, Queensland University of Technology (QUT), Brisbane, Qld 4001, Australia.

Introduction

When metal burns, it does so in the liquid phase. In promoted ignition testing, promoters are used to melt and initiate the burning of metal rod test samples. When a promoter is ignited, it transfers energy as heat to the sample, and a liquid molten drop forms at the bottom of the vertically mounted sample. While a material is burning, the molten droplets continue to form and drip away in a cyclic pattern as the rod is consumed. If conditions are not able to sustain the burning, a drip will re-solidify and the sample will be extinguished. When the promoter is ignited, a portion of the rod is heated beyond the location of the promoter. This heating is termed “sample preheating.” Although there have been earlier works researching promoter effects [4], there are not enough data to make conclusive determinations about the effect these promoters have on the rod in terms of sample preheating. It has been shown in previous studies [5–8] that metals are more flammable at elevated temperatures; therefore, it is important to understand the preheating of the rods due to the promoter when establishing burn criteria. Testing was proposed to determine the distance that the promoter preheats the metal rod prior to molten drops forming. The test sample configurations were chosen to replicate ASTM G124 [2] with the exception that thermocouples were inserted along the sample at various depths and intervals. The distance heated by the promoter just prior to promoter detachment or consumption is referred to as the promoter heat affected zone (HAZ).

Experimental

Test System

National Aeronautics and Space Administration (NASA) Johnson Space Center White Sands Test Facility (WSTF) is equipped with a promoted combustion test chamber in its Hazardous Fluids Test Area. This chamber has a maximum allowable working pressure of 79.2 MPa (11,500 psi) and is used primarily for ASTM G124 standard promoted combustion testing. The gaseous oxygen used in the chamber is sampled to ensure it conforms to the requirements of MIL-PRF-27210G [9], with a minimum oxygen concentration of 99.5 %. This system and its use are described in several previous publications [2, 3].

For the current work, one of the chamber viewports was modified to incorporate a multi-thermocouple feedthrough. This feedthrough allowed for up to twelve temperature readings at test pressures up to 34.5 MPa (5000 psi). Additionally, a high-speed camera was set up to capture the ignition event. Temperature, pressure, and high-speed video were recorded for each test. Temperature and pressure data were recorded at a 50 or 100 Hz sample rate (initial tests were recorded at 50 Hz, the remainder at 100 Hz for better temperature data resolution). Burning was observed via high-speed video, which was used to establish the event duration between the ignition of the promoter and the time the initial molten-metal drop separated from the sample or was consumed. This event duration was then synchronized with sample temperature data for each test.

Sample Configuration and Test Matrix

Test samples were configured as 305 mm (12 in.) long, 3.2 mm (0.125 in.) diameter rods. Three materials were tested: 316 stainless steel, Monel®[5] 400, and commercially pure copper. Table 1 shows the test matrix for each material. Copper was chosen as the material for the majority of the testing based on its high thermal conductivity and resistance to burning at elevated pressures and concentrations of oxygen.

[5] Monel® is a trademark of Inco Alloys International, Inc., Huntington, West Virginia.

TABLE 1—*Sample configuration and test matrix.*

Sample Material	Promoter Material[a]	Pressure MPa (psi)	Number of Tests
Copper	Magnesium	0.7 (100)	1
Monel® [b] 400	Magnesium	0.7 (100)	1
316 SS	Magnesium	0.7 (100)	1
Copper	Pyrofuze®[c]	3.5 (500)	1
Copper	Magnesium	3.5 (500)	4
Copper	Aluminum	3.5 (500)	4
Copper	Magnesium	6.9 (1000)	3
Copper	Aluminum	6.9 (1000)	3
Copper	Magnesium	34.5 (5000)	4
Copper	Aluminum	34.5 (5000)	3

[a] Magnesium and aluminum promoters were initiated using Pyrofuze wire.
[b] Monel® is a trademark of Inco Alloys International, Inc., Huntington, West Virginia.
[c] Pyrofuze® is a registered trademark of Sigmund-Cohn Company, Mount Vernon, New York.

All test samples were instrumented with Type-K bare-wire beaded-type thermocouples. After the thermocouple holes were drilled, samples were washed in a liquid detergent bath at 49–66 °C (120–150 °F), immersed for five minutes, agitated for one minute, and scrubbed with a nylon brush as required. They were then agitated for two minutes with hot, deionized water until no visual evidence of detergent solution was evident, and blown dry with gaseous nitrogen. Thermocouple lead wires were 0.13 mm (0.005 in.) in diameter. Typical samples had ten thermocouples staked into 0.254–0.381 mm (0.010–0.015 in.) diameter holes with a depth of 0.254–0.508 mm (0.010–0.020 in.). Thermocouples were peened in place on the test sample rods as illustrated in Fig. 1. Spacing of thermocouples was typically as shown in Fig. 2, with thermocouple placement distances measured from the bottom of the rod. Some test samples varied in that thermocouples holes were drilled at a depth of 1.27–1.78 mm (0.050–0.070 in.). This was done to investigate the temperature deviation over the cross section of the rod. The length of engagement of the promoter onto the bottom of the rod varied. This was not by design, but rather by observation, after several tests had been conducted. As this deviation could affect test results, promoter engagement length was noted for each sample thereafter.

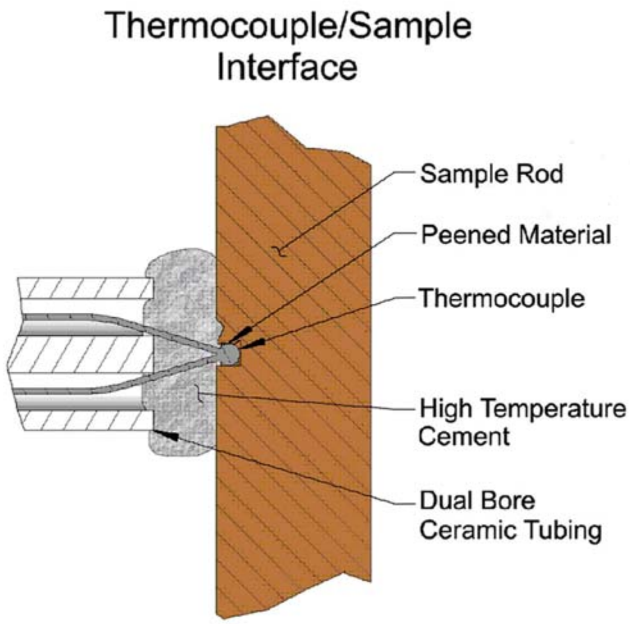


FIG. 1—*Thermocouple sample interface.*

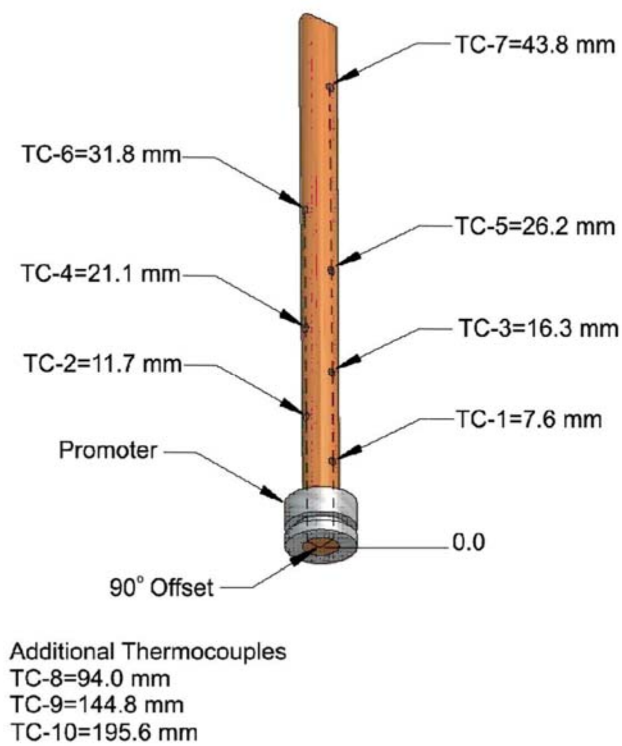


FIG. 2—*Thermocouple spacing along test sample.*

As shown in Fig. 3, dual-bore ceramic tubing and ceramic cement were used on the lower seven thermocouples to protect them during ignition of the promoter. Thermocouples TC-1 through TC-4 had a temperature range of 0–1000 °C (32–1832 °F). Thermocouples TC-5 through TC-7 had a temperature range of 0–500 °C (32–932 °F). Although this scheme worked well, it was a result of thermocouple module (isolation and amplification) availability and was not a test requirement.

wstf1206e08762

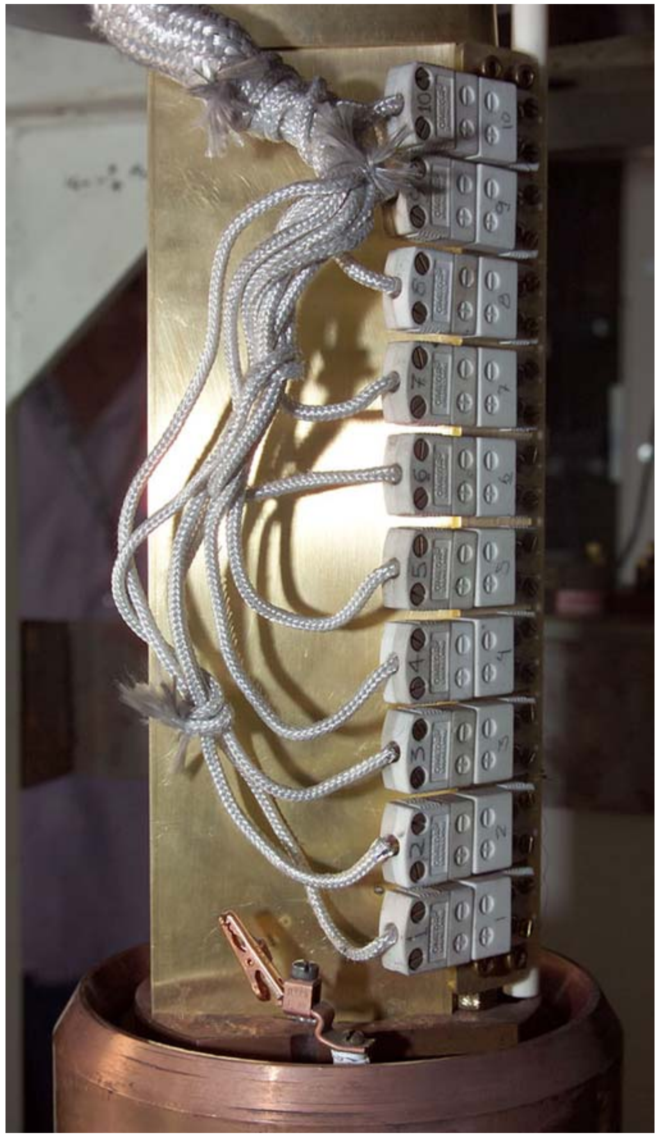
FIG. 3—*Ceramic shielding on lower seven thermocouples.*

Test Procedure

Each sample was mounted to the sample support, and the thermocouple leads terminated at the appropriate female thermocouple plug. These female plugs were vertically mounted on the sample stand and numbered 1 through 10 to mate to corresponding data channels. The sample or promoter was then wound with eight wraps of Pyrofuze, inserted into the test chamber, and the thermocouple and Pyrofuze wires were appropriately terminated. Nextel®[6] ceramic fabric sleeves were used to shield the thermocouple insulation from thermal damage including igniting and burning. A brass plate was also used between the sample and the shielded thermocouple wires for further protection. Thermocouple plug connections and shielding measures are shown in Fig. 4.

The chamber was closed and purged three times with test atmosphere at 3.5 MPa (500 psi) to remove any residual gasses. The chamber was then pressurized to the desired test pressure. Temperature and pressure data were recorded along with high-speed video.

[6] Nextel® is a trademark of 3M Company, St. Paul, Minnesota.



wstf1206e08766

FIG. 4—*Thermocouple plug connections and ignition shielding.*

Test Results

High-speed video was used to determine the event duration, i.e., the duration from the ignition of the Pyrofuze igniter wire to the first significant drip of molten metal, promoter detachment, or consumption of the promoter (if the promoter did not drop off the sample). Since no appreciable energy was added to the sample after the end of the event duration, it was assumed that the sample would ignite and begin burning or quench and solidify after this event. The event duration is thus considered to be the controlling time used when assessing the temperature data from the thermocouples. To correctly plot event duration on a time vs. temperature graph, thermocouple response lag time was considered and compensated for. Figures 5 through 7 show typical time vs. temperature plots for copper samples with aluminum promoters, with the beginning and end of the event duration shown as vertical lines. Test data in its entirety is published in a WSTF Special Test Data Report [10].

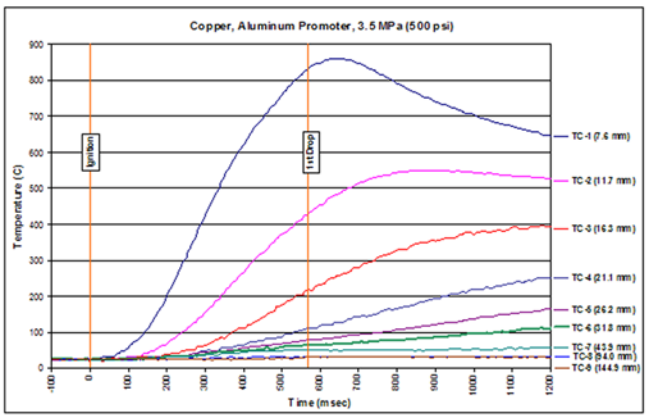


FIG. 5—*Copper sample with an aluminum promoter in gaseous oxygen at 3.5 MPa.*

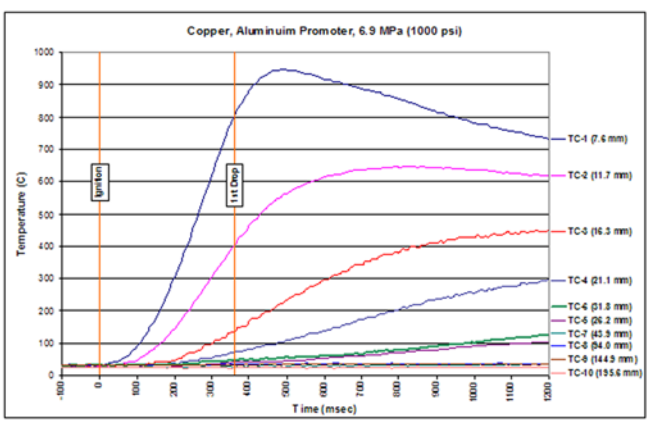


FIG. 6—*Copper sample with aluminum promoter in gaseous oxygen at 6.9 MPa.*

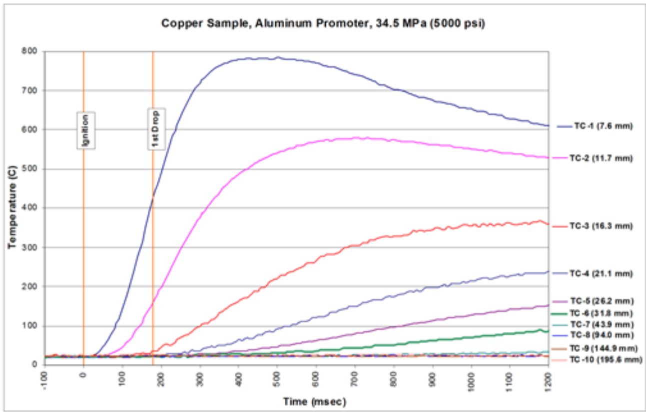


FIG .7—*Copper sample with an aluminum promoter in gaseous oxygen at 34.5 MPa.*

Initial tests exhibited some irregularities in the temperature data. Mainly, TC-1 and TC-10 showed unrealistic oscillations and negative temperature readings. In subsequent tests, these irregularities were corrected by electrically isolating the samples from the sample stand.

Discussion and Application of the Data

Although the majority of the test samples were copper rods, general observations can be made with regard to other test materials. As expected, due to their lower thermal conductivity, 316 stainless steel and Monel 400 samples exhibited a less severe promoter HAZ (Table 2). It can be seen that the event durations for the stainless steel and Monel samples are larger than the copper sample (for a given pressure), giving the promoter more time to transfer heat to the sample. However, the copper sample still exhibited higher temperatures along TC-2 through TC-5. The high temperature for TC-1 of the Monel sample is most likely due to the convective effects of the initial ignition of the promoter. Theoretical heat transfer studies of the promoter HAZ have also been investigated [11] and, as expected, complement and agree with this experimental data.

TABLE 2—*Material temperature comparison at 0.7 MPa.*

Promoter Material	Magnesium		
Pressure (MPa)	0.7		
Sample Material	316 SS	Monel® 400	Copper
Event Duration: Ignition to Drop (msec)	1050	1670	700
Thermocouple Placement (mm)	Drop Point Temperatures (°C)		
TC-1 (7.6)	—	937	732
TC-2 (11.7)	124	247	408
TC-3 (16.3)	78	108	248
TC-4 (21.1)	60	71	107
TC-5 (26.2)	52	58	67
TC-6 (31.8)	50	54	54
TC-7 (43.8)	43	43	42
TC-8 (94.0)	41	35	35
TC-9 (144.8)	31	32	31
TC-10 (195.6)	37	33	30

As expected, it was also observed that promoters burn more slowly and exhibit longer resident times when ignited at lower pressures (Table 3). This results in higher sample temperatures at the end of the event duration. Conversely, at elevated pressures promoters burn and detach from the sample much more quickly and have less of a preheating effect.

TABLE 3—*Average event duration with respect to test pressure.*

Pressure MPa (psia)	Duration (msec)
3.5 (500)	446
6.9 (1000)	328
34.5 (5000)	140

To determine the promoter HAZ, it must be understood what amount of sample preheating, in terms of temperature, is necessary for burning characteristics to be affected. Data from high-temperature promoted ignition testing [5] suggests that for several 300-series stainless steels, samples heated to 260 °C (500 °F) and below show little or no variation in flammability when compared to ambient temperature testing. Changes in flammability would be noted by changes in either the pressure where a metallic sample will support burning, or the rate at which the sample melts while it is burning (both are indicators

of metals flammability and are used historically to compare metals). Therefore, 260 °C (500 °F) was chosen as a reasonable temperature to use in determining what length of the sample was within the HAZ. The HAZ is considered the length of the rod measuring temperatures greater than or equal to 260 °C (500 °F) just as the ignition promoter is detaching from the test sample. Determining the location of this temperature condition allows accurate delineation of the HAZ within the rod and the production of burn criteria beyond which there are no further effects of the ignition promoter on the burning of the test sample.

Temperatures at the end of the ignition events were used to determine the location, from the bottom of the rod, where the samples reached 260 °C (500 °F). For each test pressure series, this location was compiled and standard deviations applied (see Fig. 8). This shows that, including standard deviation error bars, the promoter HAZ does not extend beyond 17.8 mm (0.70 in.) above the end of the rod. It should be noted that this distance includes the promoter engagement distance, which is not included when reporting burn lengths as instructed in ASTM G124. It was also observed that promoter material (aluminum or magnesium) did not greatly affect the length of the HAZ produced within the test sample (for the conditions of these tests).

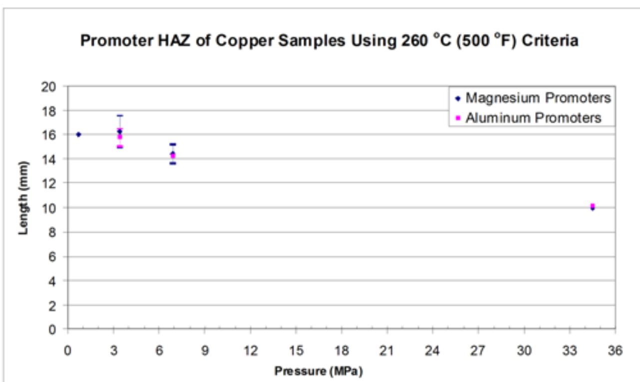
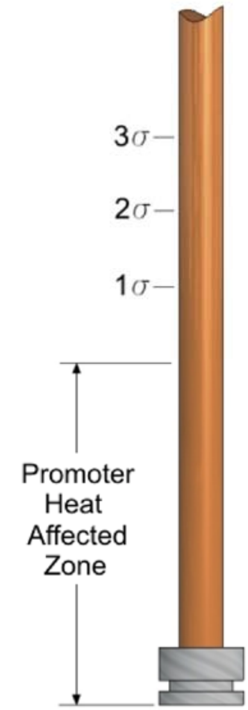


FIG .8—*Distance along rod where 260 °C was reached at promoter detachment at various test pressures.*

The mean value and standard deviation between the test pressure group with the largest promoter HAZ was then found using the Central Limit Theorem (CLT), and a three sigma distribution was applied. Using the CLT assumes there is a normal data distribution, and thus ~ 99.7 % of the data values will be

within three standard deviations of the mean. Using the most severe condition in terms of heat generation/conduction (3.5 MPa (500 psi)), and combining both aluminum and magnesium promoter sample data, yields a mean HAZ distance of 16 mm (0.63 in.) with a standard deviation of 1 mm (0.041 in.). Applying three standard deviations results in a promoter HAZ of 19 mm (0.75 in.) from the bottom of the rod (Fig. 9).

Based on these data, which have also been validated by thermal modeling [11], it was discussed and decided within NASA that samples which burn more than 30 mm (1.18 in.) above the promoter will be considered a burn [1]. To come to this length of 30 mm (1.18 in.) rather than the promoter-included length of 19 mm (0.75 in.), as determined in testing, roughly 50 % of the actual HAZ was added as a conservative buffer. This ensures all metals that burn a distance greater than 30 mm (1.18 in.) are burning independently of any promoter effects that preheated the test sample during the ignition process. For metallic materials that have a lower thermal conductivity than copper (that is, most metals), burning a distance greater than 30 mm (1.18 in.) is a large distance beyond the HAZ, and clearly the metals are burning. ASTM G124 is currently being revised and will incorporate these same pass/fail criteria.



Three Sigma HAZ 260 °C / 3.5 MPa	
HAZ (mm)	16.1
Sigma (mm)	1.0
HAZ+3(Sigma) (mm)	19.2

FIG. 9—*HAZ produced from ignition promoter after applying the Central Limit Theorem using a 260 °C (500 °F) temperature limit.*

The experimental observations and subsequent conclusion of the promoter HAZ modifies the historical views of metals flammability in the specific application of ASTM G124, as well as other tests that often require the test sample to be consumed entirely or halfway to be considered flammable. This result is also specific to the igniter materials tested. Copper was chosen as the major test material based on several factors, most significantly its resistance to burning and high thermal conductivity. A high thermally conductive material was desired to ensure effective heat transfer from the promoters, leading to large HAZs. Metals with lower thermal conductivities will have smaller HAZs. Additionally, since the promoter HAZ in this experiment is calculated using a 260 °C (500 °F) temperature bias, it should be noted that, with further research into the effects of metals flammability with respect to elevated temperature, the promoter HAZ may need to be modified accordingly. For previously tested materials where burn lengths were recorded, these burn criteria can be easily applied. However, since these burn criteria are markedly different from the previous criteria, flammability of many materials may no longer

be defined. As the required number of tests to rank a material are no longer met, additional testing may be needed. ASTM requires five no-burn tests for a material to be considered nonflammable, NASA requires ten [1, 2]. Additionally, after applying these burn criteria to existing material data, many previously considered metals have not been tested at a nonflammable condition. A small sample set (see Table 4) shows some metals that are now considered flammable at pressures that were previously considered nonflammable, and consequently there are insufficient data to establish a nonflammable condition without further testing.

TABLE 4—*Flammability of select metals using 30 mm burn criteria.*

Material	Nonflammability Pressure using Previous Criteria MPa (psia) [12]	Flammability Pressures using 30 mm Criteria MPa (psia) [13]
Haynes®[a] 188	20.7 (3000)	Flammable at 20.7 (3000), no lower pressures tested
Hastelloy®[a] C22	20.7 (3000)	Flammable at 17.2 (2500), no lower pressures tested
Elgiloy®[b]	10.3 (1500)	Flammable at 10.3 (1500), no lower pressure tested
Inconel®[c] 718	5.2 (750)	Flammable at 3.5 (500), no lower pressure tested
17-4 PH SS	3.5 (500)	Flammable at 3.5 (500), no lower pressure tested

[a] Haynes® and Hastelloy® are registered trademarks of Haynes Stellite Company, Kokomo, Indiana.
[b] Elgiloy® is a registered trademark of Elgin National Watch Company, Elgin, Illinois
[c] Inconel® is a registered trademark of Inco Alloys International, Huntington, West Virginia.

Conclusion

The goal of this testing was to characterize the heating effects of an ignition promoter on a copper sample as is typically configured in promoted combustion flammability testing. Furthermore, the goal was to apply these findings to empirically establish better suited flammability criteria that incorporate the heating effect of the ignition promoter on the test sample. It was determined that the largest HAZ produced will likely be approximately 19 mm long (or less) on a copper rod, and if a test sample burns a distance longer than this, it is clearly burning independently of the ignition-promoter effect. The success of this testing allows scientific burn criteria to be established within NASA standards, with other international standards soon to follow suit. In accomplishing this goal, there were many observations that aid in the understanding of the intricacies of promoted combustion testing (e.g., ignition event duration

with respect to test pressure, the severity of magnesium and aluminum promoters on the test sample with respect to the gaseous oxygen environment).

References

[1] NASA Technical Standard NASA-STD-(I)-6001A. Flammability, Offgassing, and Compatibility Requirements and Test Procedures, NASA Headquarters, Washington, D.C., 2008.

[2] ASTM G124. Standard Test Method for Determining the Combustion Behavior of Metallic Materials in Oxygen-Enriched Atmospheres, *ASTM Standards Related to Flammability and Sensitivity of Materials in Oxygen-Enriched Atmospheres,* ASTM International, West Conshohocken, PA, 1995.

[3] “Determination of Upward Flammability of Materials in Pressurized Gaseous Oxygen or Oxygen-enriched Environments,” Part 4 of ISO 14624-4:2003, *Space Systems – Safety and Compatibility of Materials*, International Organization for Standardization (ISO), 2003.

[4] De Wit, J. R., Steinberg, T. A., and Stoltzfus, J. M., “Igniter Effects on Metals Combustion Testing,” *Flammability and Sensitivity of Materials in Oxygen-Enriched Atmospheres: Ninth Volume, ASTM STP 1395*, T. A. Steinberg, B. E. Newton, and H. D. Beeson, Eds., ASTM International, West Conshohocken, PA, 2000.

[5] Slockers, M. J., and Robles-Culbreth, R., “Ignition of Metals at High Temperatures in Oxygen,” *Flammability and Sensitivity of Materials in Oxygen-Enriched Atmospheres: 11th Volume, STP 1479,* D. B. Hirsch, R. Zaweirucha, T. A. Steinberg, and H. M. Barthelemy, Eds., ASTM Internationanl, West Conshohocken, PA, 2006, pp. 62–79.

[6] Engel, C. D., Herald, S., and Davis, E., “Promoted Metals Combustion at Ambient and Elevated Temperatures,” *Flammability and Sensitivity of Materials in Oxygen-Enriched Atmospheres: 11th Volume, STP 1479,* D. B. Hirsch, R. Zawierucha, T. A. Steinberg, and H. M. Barthelemy, Eds., ASTM International, West Conshohocken, PA, 2006, pp. 51–61.

[7] Zaweirucha, R., and Million, J. F., “Promoted Ignition-Combustion Behavior of Engineering Alloys at Elevated Temperatures and Pressures in Oxygen Gas Mixtures” *Flammability and Sensitivity of Materials in Oxygen-Enriched Atmospheres: Ninth Volume, STP1395*, T. A. Steinberg, B. E. Newton, and H. D. Beeson, Eds., ASTM International, West Conshohocken, PA, 2000.

[8] Sato, J., and Hirano, T., “Behavior of Fires Spreading Along High-Temperature Mild Steel and Aluminum Cylinders in Oxygen,” *Flammability and Sensitivity of Materials in Oxygen-Enriched Atmospheres: Second Volume, STP 910*, M. A. Benning, Ed., ASTM International, Philadelphia PA, 1986, pp. 118–134.

[9] MIL-PRF-27210G, “Oxygen, Aviator’s Breathing, Liquid and Gas,” Military Performance Specification, United States Department of Defense, Washington, D.C., 1997 or most current revision.

[10] Stoltzfus, J., and Sparks, K. “Promoted Combustion Promoter Heat Affect Zone Testing.” Special Test Data Report WSTF # 08-43113. NASA Johnson Space Center White Sands Test Facility, Las Cruces, NM, Publication in progress.

[11] Steinberg, T., Lynn D., Sparks, K., Stoltzfus, J., Smith, S., “Defining Self-Sustained Burning of Cylindrical Metal Rods through Characterization of the Thermal Effects of the Ignition Promoter,” *Flammability and Sensitivity of Materials in Oxygen-Enriched Atmospheres: Twelfth Volume, ASTM STP* (publication in progress).

[12] Beeson, H., Stewart, W., Woods, S., *Safe Use of Oxygen and Oxygen Systems: Handbook for Design, Operation, and Maintenance,* ASTM International, West Conshohocken, PA, 2000.

[13] Beeson, H., Smith, S., Stewart, W., *Safe Use of Oxygen and Oxygen Systems: Handbook for Design, Operation, and Maintenance: 2nd Edition*, ASTM International, West Conshohocken, PA, 2007.